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DEPARTMENT OF TRADE
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This is to certify that this is a true copy of the provisional specification filed in support of South

African Patent Application No. 99/5746 entitled BIOLEACHING OF SULPHIDE MINERAL

CONCENTRATES on 7 September 1999

15/3

PRIORITY DOCUMENT

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Geteken te Signed at PRETORIA in die Republiek van Suid-Afrika, hierdie in the Republic of South Africa, this 12th dag van day of

September 2000

Registrateur van Patente
Registrar of Patents

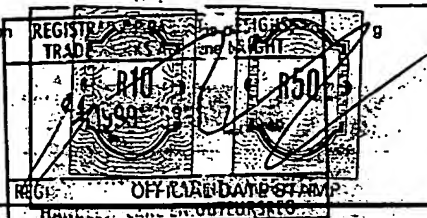
RECEIPT

(Section 30(1) - Regulation 22)

The grant of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate.

OFFICIAL APPLICATION NO.

21 01 995746



FULL NAME(S) OF APPLICANT(S)

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BILLITON INTELLECTUAL PROPERTY B.V.

ADDRESS(ES) OF APPLICANT(S)

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TITLE OF INVENTION

54

BIOLEACHING OF SULPHIDE MINERAL CONCENTRATES

Priority is claimed as set out on the accompanying Form P2.

The earliest priority claimed is: NONE

This application is a patent of addition to Patent Application No.

21 01

This application is a fresh application in terms of section 37 and based on Application No.

21 01

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1 A single copy of a provisional specification of10..... pages
- ☐ 2 Two copies of a complete specification of pages
- ☒ 3 ...1..... sheet of Informal Drawings
- ☐ 4 sheets of Formal Drawings
- ☐ 5 Publication particulars and abstract (Form P8 in duplicate)
- ☐ 6 A copy of Figure of drawings (if any) for the abstract
- ☐ 7 Assignment of Invention
- ☐ 8 Certified priority document(s) Number(s)
- ☐ 9 Translation of priority document(s)
- ☐ 10 An assignment of priority rights
- ☐ 11 A copy of the Form P2 and the specification of SA Patent Application
- ☐ 12 A declaration and power of attorney on Form P3
- ☐ 13 Request for ante-dating on Form P4
- ☐ 14 Request for classification on Form P9
- ☒ 15 Form P2 in duplicate

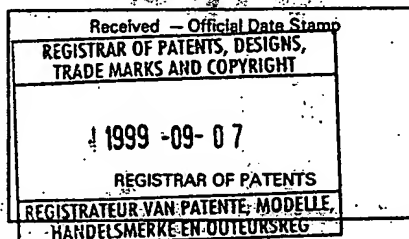
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Dated this 7th day of SEPTEMBER 1999

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PATENT AGENTS FOR APPLICANT(S)



REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

PROVISIONAL SPECIFICATION

(Section 30(1) - Regulation 27)

OFFICIAL APPLICATION NO

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| 21 | 01 | 995746 |
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LODGING DATE

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| 22 | 7 SEPTEMBER 1999 |
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FULL NAME(S) OF APPLICANT(S)

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| 71 | BILLITON INTELLECTUAL PROPERTY B.V. |
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| 72 | DAVID WILLIAM DEW and PETRUS BASSON |
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TITLE OF INVENTION

| | |
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| 54 | BIOLEACHING OF SULPHIDE MINERAL CONCENTRATES |
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5 The dissolved oxygen concentration may be controlled by controlling the addition of oxygen to the solution. The oxygen may be added by feeding a stream of air, oxygen-enriched air, or substantially pure oxygen to the reactor.

10 The temperature at which the bioleaching step is carried out, and the parameters of the predetermined range of dissolved oxygen concentration, depend at least on the strain or strains of bacteria used in the bioleaching step.

In a general sense a more efficient bioleaching step is carried out when the temperature is increased but a high temperature on the other hand can give rise to material or engineering problems.

15 If the dissolved oxygen concentration in the solution is too high then the bacteria population growth will be limited or prevented. On the other hand if the dissolved oxygen concentration is too low then the bacterial population growth is inhibited.

20 A preferred temperature range for carrying out step (a) is from 60°C to 85°C.

A preferred range of dissolved oxygen concentration is from $0,5 \times 10^{-3} \text{ kg/m}^3$ to $2,0 \times 10^{-3} \text{ kg/m}^3$. It is to be noted nonetheless that at least the upper limit of the dissolved oxygen concentration depends on the microorganism used in the

25 bioleaching process.

5 Merely by way of non-limiting examples moderate thermophiles of the type *Sulfobacillus* can be used in step (a) operating at a temperature of up to 65°C. Thermophiles of the type *Sulfolobus*, e.g. *Sulfolobus metallicus*, can be used for operation at temperatures of from 60°C to at least 85°C.

10 The method may include the step of adding carbonaceous material to the solution in the reactor to maintain cell growth within the reactor. Thus carbon dioxide may for example be added to the reactor or carbonate minerals may be added to the reactor or to the feed of the concentrate to the reactor.

15 BRIEF DESCRIPTION OF THE DRAWING

The invention is further described by way of example with reference to the accompanying drawing which is a schematic representation of a portion of a bioleaching plant in which the method of the invention is carried out.

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DESCRIPTION OF PREFERRED EMBODIMENT

The accompanying drawing illustrates a bioleaching plant in which the method of the invention is practised and which includes a bioreactor 10, an agitator or

25 mixer 12, a probe 14 for measuring the dissolved oxygen concentration in a mineral concentrate slurry 16 in the reactor 10, a probe 18 for measuring the

5 dissolved carbon dioxide concentration in the solution 16, a source 20 of
oxygen, a source 22 of carbon dioxide, control valves 24 and 26, and a sparger
28.

10 The drawing does not illustrate steps which are carried out before or after the
bioleaching phase, in order to recover metal content from the concentrate.
These aspects are known in the art and play no role in an understanding of the
method of the invention.

15 The bioleaching of a sulphide concentrate at an elevated temperature results in
a high rate of sulphide mineral oxidation but is dependent on the supply of
oxygen and carbon dioxide at adequate rates. The absorption of oxygen and
carbon dioxide in the reactor 10 is limited in each case by the rate of mass
transfer from the gas phase into the solution phase. For oxygen the rate of
oxygen absorption is defined by the following equation:

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$$R = M. (C^* - C_L)$$

where R = Oxygen demand as mass (kg) per unit volume (m^3) per unit
time(s) ($kg/m^3/s$)

25 M = Oxygen mass transfer coefficient in reciprocal seconds (s^{-1})

C^* = Saturated dissolved oxygen concentration in mass (kg) per
unit volume (m^3) (kg/m^3)

30 C_L = Dissolved oxygen, concentration in solution as mass (kg)
per unit volume (m^3) (kg/m^3)

The factor $(C^* - C_L)$ is referred to as the oxygen driving force.

5 A similar equation may be used to describe the rate of carbon dioxide supply to the solution.

If the sulphide mineral oxidation rate is increased the oxygen demand increases

10 proportionately. To meet a higher oxygen demand either the oxygen mass transfer coefficient (M) or the oxygen driving force ($C^* - C_L$) must be increased.

An increase in the oxygen mass transfer coefficient may be achieved by increasing the power input to the mixer 12. This improves gas dispersion in the concentrate 16. However an increase in the oxygen mass transfer coefficient of 15 50% requires an increase in the power input to the mixer by a factor of as much as 200% with a commensurate increase in operating costs.

The oxygen driving force may be increased by increasing the saturated dissolved oxygen concentration C^* and reducing the dissolved oxygen 20 concentration C_L .

Microbial population growth is limited or prevented if the dissolved oxygen concentration C^* reaches too high a level. A concentration level in the range of from $4 \times 10^{-3} \text{ kg/m}^3$ to $8 \times 10^{-3} \text{ kg/m}^3$ has been found to be detrimental to

25 *Sulfolobus* microbial strains. Certain other strains however have been found to be tolerant to dissolved oxygen concentrations of up to $10 \times 10^{-3} \text{ kg/m}^3$.

The lower limit for the dissolved oxygen concentration is in the range of from 0,5 x 10⁻³ kg/m³ to 2.0 x 10⁻³ kg/m³ for *Thiobacillus* or *Sulfolobus* microbial strains.

It is apparent, therefore, that it is essential to monitor the dissolved oxygen

concentration in the concentrate slurry 16 and this is done by means of the

probe 14 which provides a control signal to a control device 30 which in turn

controls the operation of the valve 24. As has been indicated in the preamble to

this specification the rate of sulphide mineral oxidation which can be achieved

when operating at a relatively low temperature of the order of from 40°C to 55°C

is limited. In order to increase the rate of oxidation it is desirable to make use of

thermophiles and to operate at temperatures of from 60°C to 85°C. Any suitable

bacterium or archaea capable of operating within this temperature range may

be used. The optimum operating temperature is dependent on the genus and

type of microorganism used. Thus moderate thermophiles of the type

Sulfobacillus are suitable for operating at a temperature of up to 65°C.

Thermophiles of the type *Sulfolobus* are suitable for operating at temperatures

of from 60°C to at least 85°C. *Sulfolobus metallicus*, for example, shows optimal

growth in the temperature range of from 65°C to 78°C.

The applicant has established that the operation of the bioleaching process at

elevated temperatures of from 60°C to 85°C with oxygen enriched air enhances

the oxygen mass transfer coefficient (M) significantly compared to a bioleach

5 operation carried out at a temperature of from 40°C to 45°C with air. For example, typical design oxygen mass transfer coefficients for bioleaching with air at 40°C to 45°C, in commercial bioreactors (greater than or equal to 100m³ in volume) are of the order of 0,035 s⁻¹. In one experiment with the method of the

10 invention and using the source 20 to supply oxygen to the solution 16 in a controlled manner, taking into account the criteria already mentioned, and conducting the bioleaching step at a temperature of the order of 78°C, oxygen mass transfer coefficients in the range of from 0,08 s⁻¹ to 0,09 s⁻¹ have been attained. This enhancement is higher than expected and cannot be ascribed to the effect of temperature alone, for an increase in temperature, from 45°C to 15 78°C, would only be expected to improve the oxygen mass transfer coefficient from a value 0,035 s⁻¹ to a value of 0,055 s⁻¹. As stated, the coefficient increased to 0,09 s⁻¹ which is about 60% greater than the expected value of the coefficient (0,055 s⁻¹) which is attributable only to the higher operating temperature.

20 The addition of oxygen to the concentrate 16 must be controlled in order to maintain the minimum dissolved oxygen concentration in solution at a value of from 0,5 x 10⁻³ kg/m³ to 2,0 x 10⁻³ kg/m³ and to ensure the dissolved oxygen concentration does not exceed the upper threshold value at which microbial cell growth is prevented. Again it is pointed out that the upper threshold

25 concentration depends on the genus and strain of microorganism used in the bioleaching process. A typical upper threshold value is in the range of from 4

In carrying out the method of the invention the temperature of the reactor may be controlled in any suitable way. Preferably the reactor is insulated and heating

takes place by means of energy which is released by bacterial activity i.e. the

oxidation of carbonates. The temperature of the slurry is regulated using an internal cooling system. The reactor is a closed vessel and off gas may be passed through a condenser to recover moisture from the gas.

The accompanying drawing illustrates the addition of oxygen from a source of pure oxygen. The pure oxygen can be mixed with an air stream passing through a control valve to provide an oxygen enriched air stream containing, for example, from 25% oxygen to 70% oxygen.

The controlled addition of pure oxygen or an oxygen enriched air stream directly into the bioreactor or into an airstream introduced into the bioreactor improves the oxygen utilisation efficiency. The oxygen utilisation for a conventional commercial bioleach plant (at least 100m^3 in volume) operating at from 40°C to 45°C with air may be expected to achieve a maximum oxygen utilisation of from 40% to 50%. Consequently 40% to 50% of the total mass of oxygen supplied to

the bioleach plant only is used to oxidise the sulphide minerals. With the process of the invention the oxygen utilisation is significantly higher, of the order

5 of from 60% to 95%. The higher oxygen utilisation is achieved by controlled oxygen addition and results from the enhanced oxygen mass transfer rate and by operating at low dissolved oxygen concentrations in the solution phase.

10 The probe 18 measures the carbon dioxide content in the solution and, via the valve 26, controls the addition of carbon dioxide from the source 22 to the sparger 26. Alternatively carbonate minerals may be added to the concentrate prior to the feeding thereof to the reactor, or may be added directly to the concentrate slurry in the reactor.

15 Another variation which can be adopted is to move the probe 14 to a position 14A above the concentrate slurry to measure the oxygen contained in the gas above the concentrate slurry, and to use this measurement to control the addition of oxygen to the concentrate slurry. Similarly the probe 18 may be moved to a position 18A to measure the carbon dioxide contained in the gas
20 above the concentrate slurry, and to use this measurement to control the addition of carbon dioxide to the concentrate slurry.

DATED this day

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Patent Agents for the Applicant

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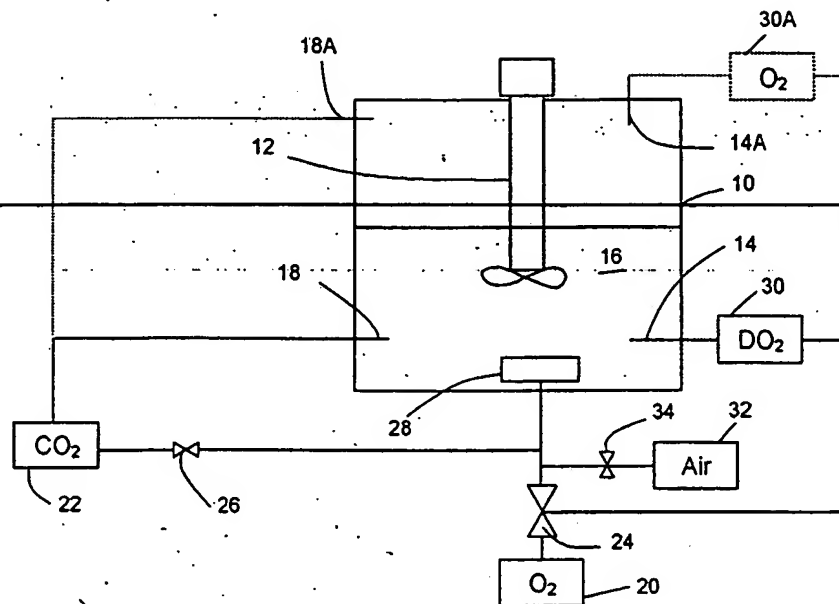
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DATED this 7th day of SEPTEMBER 1999.

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Mr. Khan